Effect of ultrasound on bovine and ovine skins soaking

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Abstract

Ultrasound as the only mechanical effect in the first soaking of bovine rawhides and goatskins were tested. Three different working systems were used at pilot plant level. In the first work system only the soaking float was subjected to the action of ultrasound. In the second and third systems, both the soaking float and the skin were underwent the same action. In the third system a surfactant was added to the soaking float. The variables analyzed were the water absorbed by the skin and its organoleptic properties, the chemical oxygen demand, the suspended solids, and the conductivity of the wastewater. Through analysis of variance in bovine hides, the influence on the results of the hide part (butt, neck or belly) and the work system employed were compared. Related to the values obtained by performing a comparative soaking on stationary way, the use of ultrasound allowed an increase of up to 23% in water absorption of the soaked skins and up to 49% of COD, 58% of SS and 34% of conductivity in soaking floats. For goatskins, due to their thickness and size, a comparison based on the work system was developed. It was proved that for dried goatskins and one of the tested systems of ultrasound application, the skin becomes saturated with water in 36% less time than if it was soaked on stationary way. With the same soaking time, the ultrasound application showed an increase of up to 16% of water absorption in soaked skins and up to 162% of COD, 87% of SS and 9% of conductivity in soaking floats. In both cases, the results were compared with those obtained when the first soaking was performed on stationary way or in drumming. The results show that the use of ultrasound in the first soaking of the skins is a valid alternative that may be useful, at the industrial level, to replace the working systems where the use of drums is not possible.

Keywords

Ultrasound; Leather processing; Skin/hide soaking; Ultrasound effect.

1. Introduction

Currently, a very important part of research in the field of tanning is focused on developing environmentally friendly processes. These new processes are intended to enable an improvement of the quality of the leathers obtained while achieving a reduction in the time, water and energy consumed, as well as pollution generated. In this vein, recent studies have been published on the ecological utilization of tannery waste [1], on the kinetics [2] and effects [3] of different enzymes in unhairing of hides and on the reuse of chrome tanning floats [4].

Soaking is the first step of the beamhouse, which is a set of operations which constitute the first part of the tanning process. In many cases soaking is divided in two stages which can be called the first and the second soaking. Depending on the conditions of the skin, the objectives of the first soaking may vary. The first soaking is used to make a first cleaning of the skin, removing a great amount of dirt and unwanted materials attached to it. Another objective of the first soaking is to increase the skin water content in order to reach more effectiveness in the subsequent cleaning. As this is a very important operation, a soaking performed with low quality standards can seriously undermine the quality of the leather obtained at the end of tanning process [5].

In many cases the soaking is performed in a drum because the mechanical effect provided assistance in accelerating the penetration of water and chemicals added to the float into the skin. However, there are some cases where the mechanical effect is not advisable. Such cases are, for example, the first soaking of dried bovine hide or the first and second soaking of sheepskin [6].

The mechanical effect can damage dried skin during the first soaking, causing excessive fiber breakage and even defects in the grain. To avoid this, it is possible to use pits or paddles in the first soaking with little or without mechanical effect. An important drawback is that a lower mechanical effect requires more time in order to obtain the same moisture and skin cleaning. An additional drawback is also the large amount of water used in the operation.

In the case of sheepskins, the mechanical effect causes felting wool. This means that the strands of wool get knotted together. Felting makes it impossible to continue the process without unhairing the skin, since it is virtually impossible to undo the knots once they have appeared. Therefore, there are certain goods (e.g. double-face) impossible to be obtained. We must also consider that even if we manage to unhair the skin and manufacture other goods, the economic damage is likely to be significant as in order to sell the wool and obtain profits, it must not be felted. The wool, which is a recoverable and valuable product, becomes a waste. There is also the option of eliminating, or drastically reducing the mechanical effect. The disadvantages are also the same as in the case of soaking dried skin: extra time and an increase of water consumption that, in the manufacture of double-face, can be about 40L of water per 1 kg of sheepskin [7].

Therefore, it seems reasonable to investigate the possibility of generating the mechanical effect by exploring an alternative system in order to overcome the drawbacks we just mentioned. The application of ultrasound in the soaking is a possible alternative to be considered. Power ultrasound can enhance a wide variety of chemical reactions and processes. This effect is due to the cavitation, which is the growth and explosive collapse of microscopic bubbles as a result of cycles of compression and rarefaction when the sound waves pass through a liquid medium. Cavitation produces remarkable mechanical and chemical effects, such as an intense agitation, dispersion, emulsification, etc.

The application of ultrasound in the tanning operations has been investigated for many years. The first documented experiments were published in 1950 [8]. In the following decades, several researchers studied the application of ultrasound in different processes related to the tanning process [9]. Technological problems prevented their application in industrial practice. However, the materials and technology used in the manufacture of ultrasound equipment have significantly improved over time. For this reason, in recent years, several research groups have become interested in the possibilities offered by this technology and the feasibility of its application in the leather field. The effect of ultrasound on the skin structure [10] and on various operations that make up the tanning process has been studied: unhairing [11], degreasing [12] , chrome tanning [13], vegetable extracts tanning [14], titanium salts tanning [15], dyeing [16] and fatliquoring [17]. The effectiveness of ultrasound use in the manufacture of vegetable extracts [18], dyes [19] and oils [20] for tanning and in the enzymatic hydrolysis of leather waste [21] has been tested. Few of these works studied the soaking operation [22]. Good results were obtained. However, these studies have been conducted with laboratory equipment, with a ratio between the volume of the float used and the electric power consumed to nonviable industrial reproduction.

The aim of our work is to take a step towards the application of ultrasound in the soaking of skins at an industrial scale. To achieve this, it is necessary to assess in which circumstances this application offers advantages versus the traditional soaking systems. These advantages may be mainly in the environmental (water saving) or economic (less processing time) aspects. To perform this assessment, it is necessary to enhance the research carried at on the subject until today. For this purpose, we conducted a set of experiments at pilot plant level, with working conditions that enabled to simulate the industrial level with reliability. Ultrasound equipment, commonly used in the industry, which can work in different ways, was used. We worked with a ratio range between the volumes of the floats used and the electric power consumed bearable to industrial reproduction. Another novelty in our research is that bovine hides were included, while in previous works only goatskins (much thinner) were tested.

2. Materials and methods

2.1. Material

Tests were performed with dried-salted bovine hide (hide thickness: approximately 3mm, weight of each hide: approximately 25 kg) and goatskin (skin thickness: approximately 1mm, weight of each skin: 2 kg approximately).

The only chemical used was an anionic surfactant: Humectol Rapid. Cromogenia Units brand.

The tests were carried out using ultrasound tubular equipment, Gescoven brand, composed of a generator, a transmitter and a stainless steel cylindrical casing.

The generator (Figure1) can deliver a maximum electrical power of 800W, which can be regulated. It can emit at four different power levels corresponding to 100%, 85%, 75% and 60% of maximum power. Frequency: 25 kHz.

The tubular transmitter (Figure 2) is 0.67 m long and 0.07 m in diameter. Its outer wall is made of stainless steel. It is connected to the generator by a coaxial cable.

The stainless steel cylindrical casing is 1.2 m long and 0.12 m in diameter. It's empty in the inside, and the bottom is covered. The upper end is hollow. The transmitter can seal this upper end. On the top and back of the side wall there are two holes for entry and exit of liquid to be sonicated.

We also used a 1m-high (i.e. diameter) and 0.4m-wide polypropylene drum, electrical power of 2000 W, Italprogetti brand as well as a submersible water pump (approximate flow: 40L/min), Leader brand.

2.2. Working Systems

Tests were performed with different degrees of mechanical effect: With totally static (no mechanical effect) floats, with the mechanical effect provided by ultrasound and with the mechanical effect provided by the drum rotation.

Tests with ultrasound were performed in three different ways. In all cases the skin was immersed in a water float inside the stopped drum.

In the first case, named “External System”, a pump was submerged in the first soaking float that was inside the drum. The pump sucked up the float through a hose to the cylindrical casing containing the ultrasound transmitter. Then, the float would return to the drum after being subjected to the action of ultrasound for a specified period of time. In this case, the skin was soaked with water that had been previously subjected to the action of ultrasound. Thus, the skin did not directly undergo the effect of ultrasound. Figure 3 represents a diagram of the system and Figure 4 is a photo of the system.

In the second case, named “Direct System”, the transmitter was directly immersed in the float and the ultrasound acted on the float and on the skin at the same time. Figure 5 represents a diagram of the system and Figure 6 is a photo of the system.

In the third case, named “Surfactant System”, ultrasound acted in the same way as in the second case, except for the fact that a surfactant was added to the float.

These three work systems were chosen for two reasons. The first was to test whether the direct action of ultrasound on the skins may improve soaking. The second was to test whether the addition of surfactant in the float may improve the soaking of the skins.

2.3. Studied variables

In the case of bovine hides, tests were performed to find out the influence of two variables on different properties of the hide and also on the resulting float. The two variables and the different levels of each parameter tested were the following: Part of the hide (belly, neck or butt) and working system (the three systems are explained in Section 2.2).

For goatskins only the effect of the working system was studied as the thinness of the skins caused their water saturation to be very fast. Therefore it made no sense to try to assess differences between each part of the skin.

All the results were subjected to variance analysis.

2.4. Methodology

2.4.1. Fixed working conditions

Float volume was 200L for controls and 100L for tests using ultrasound. Lower volumes are not sufficient because neither the transmitter nor the submersible pump would work properly.

Preliminary tests to determine the power used and the time of ultrasound application were carried out. For each working system, the rise of temperature in the float as a function of time using ultrasound was studied. Results indicated that to avoid an excessive float temperature increase, power should be the minimum available, 480W. The average temperature in all tests was 27 °C.

2.4.2. Tests on bovine hides

All the tests on bovine hides lasted two hours.

Five different working systems were tested: the three systems using ultrasound, the static system and the system using the drum to provide the mechanical effect.

In the working system named "Surfactant System", 0.5 g/L of surfactant was added to the float. In all other tests no chemicals were added to the float.

Each part of the hide (belly, neck or butt) was cut into two parts. One half was soaked with the aid of ultrasound and the other half was soaked statically.

When the transmitter was out of the drum, inside the steel casing, the use of ultrasound for 30 minutes was alternated with a rest period of 30 minutes. This sequence was repeated again. The total soaking time was two hours, but ultrasound only worked one hour. To prevent excessive increases in float temperature, 30 minutes was the maximum time of continuous ultrasound use.

With the transmitter directly immersed in the soaking float, the total time of ultrasound application was one hour. Ultrasound was applied continuously, without any rest, because of the slow increase in float temperature. Then, the hide rested another hour in the soaking float without any mechanical effect.

For the drum soaking, the running time was two hours. Only half butt was soaked. The other half butt was soaked statically.

2.4.3. Tests on goatskins

The main differences versus bovine hide tests were:

- Each goatskin was divided into two pieces along the backbone. One half was soaked by one of the three soaking systems with ultrasound and the other half was soaked with no mechanical effect. Goatskins weight was 10kg for all the tests.

- Soaking times were lower. The goatskins appearance was regularly checked and each soaking was adjusted depending on the time in which the experts considered that the goatskins were already completely soaked and the time that ultrasound could be running without float overheating. For each work system, time was different. Table 1 shows time spent on each work system.

All soaking tests without mechanical effect had the same duration: 70 minutes.

- A test was run with the working system in which the ultrasonic transmitter is placed inside the cylindrical steel casing in order to compare the water saturation rate on the goatskins depending on the work system used in the soaking (with ultrasound or statically).

2.4.4. Floats and skins analyses

The analyses performed on the soaking floats for each of the tests were Chemical Oxygen Demand (COD), Suspended Solids (SS) and Conductivity. Analyses were carried out according to the Standard Methods [23].

COD from each starting soaking float with surfactant was determined. The results were subtracted from the COD values obtained in the analysis of the final soaking floats containing surfactant. Thus, these results were comparable with those of the floats without surfactant.

The amount of water absorbed by the skin was also determined. There is no official method for this determination. The amount of water absorbed by the skin was calculated with the following formula (1):

$$\%\ H_2O = 100\ (W_f - W_o)/W_o \qquad (1)$$

W_0 is the skin weight before soaking and W_f is the skin weight after soaking.

This is an approximation. However, it enables us to determine the cases in which there is a clear water absorption difference between various skins.

Finally, at the end of each test, an organoleptic control of the soaked skins was carried out by a group of experts in order to check their soaking degree.

3. Results

3.1. Tests on bovine hides

Organoleptic controls confirmed that bovine hides were only partially soaked as expected given their significant thickness, No differences were detected between the quality of the hides soaked with the use of ultrasound and the quality of the hides soaked in static without the use of ultrasounds.

Results are shown in Table 2.

Analyses of variance for each of the two variables studied were carried out. The analyses enabled us to identify the significant differences in results depending on the part of the hide soaked or the working system used to soak with the aid of ultrasound.

Table 3 shows the analysis of variance of COD results and Figure 7 is a graphical representation of the same analysis of variance depending on the working system being used.

The P-Value column enables us to know whether the results obtained by carrying out the different tests are significantly different or not. When the variable "Work System" was analyzed, the result was 0.0267. This means that at least one of the three systems tested yielded different results compared to the other two. The significance level was calculated with the following formula (2):

$$\% \text{ Signification level} = 100\ (1 - \text{P-Value}) \qquad (2)$$

In this case, the significance level was over 97%.

In Figure 5 the three vertical lines represent the COD ranges of values depending on the Work System used. There was no difference between the results obtained with the Direct System and the Surfactant System (the lines are at the same level). In contrast, the results obtained using the External System were significantly lower (as its line is below).

The combined interpretation of the values of P-Value column in Table 3 and in Figure 5 enables us to conclude that when ultrasound affected only the float, significantly less amount of COD was obtained versus the other two working systems studied, in which ultrasound affected both the float and the hide. The significance level was over 97%. In contrast, the results showed no significant differences between the other two work systems.

The ANOVA yielded the following conclusions:

- No significant differences were found in water absorption both in the working system and the part of the hide used.

- Higher values of conductivity, COD and SS, were obtained by soaking the butt than by soaking the belly or the neck.

- Conductivity values were not significantly different depending on the working system.

- Referring to COD and SS, higher values were obtained in the work systems where the ultrasound transmitter was directly submerged in the float soaking (Direct and Surfactant systems).

The results obtained in soakings performed with the aid of ultrasound were compared with the ones obtained with no mechanical effect. Better results, in practically every case, were obtained when ultrasound was applied. The improvements greatly varied depending on the hide part soaked and depending on the ultrasound system application (e.g., COD variation ranged between 8.1% and 49.3%). The use of ultrasound enables an increase of up to 23% in water absorption of the soaked skins and up to 49% in COD, 58% in SS and 34% in conductivity of the soaking floats.

Finally, the results obtained by soaking butts using ultrasound were compared with those using the drum. Results obtained both in the float conductivity and in water absorbed by the hide were similar. COD obtained using the drum (8.53 kg COD/t hide) was significantly lower than the results obtained in soaking with the Direct or the Surfactant systems, but higher than those obtained using the External system. SS obtained using the drum (9.02 kg SS/t hide) was higher than the results obtained using ultrasound.

3.2. Tests on goatskins

As explained in the methodology section (2.4), the soaking time in the tests on goatskins using ultrasound was different depending on the working system applied. To better understand the effect of the working system in each analyzed parameter, the percentage difference between results obtained in the tests conducted with ultrasound and the test performed without mechanical effect was calculated. Table 4 shows the percentage results obtained for COD and SS.

Results show that soaking with the ultrasound transmitter directly submerged in the float containing surfactant is the most effective working system, followed by the same system without surfactant in the float. Therefore, results showed that soaking with the ultrasound transmitter submerged in the cylindrical casing is the less effective system. Results of COD and SS obtained for the three systems show dramatic increases compared with those obtained by performing soaking without ultrasound.

These results seem quite reasonable, as they confirm that both the surfactant presence in the float and the ultrasound action on the goatskin increase the cleanliness.

The time which the goatskin reaches the proper degree of soaking (maximum water absorption) was determined by a joint assessment of the water absorption rate and the organoleptic control performed on the goatskins every 5 minutes. Soaking without ultrasound aid lasted 55 minutes and soaking with the ultrasound transmitter immersed in the cylindrical casing lasted 35 minutes. Therefore, time saving is approximately 36%.

No differences were detected between the quality of the skins soaked with the use of ultrasound and the quality of the skins soaked in static without the use of ultrasound.

4. Discussion

The assessment of all the results of the tests on bovine hides (section 3.1) shows that the use of ultrasound in bovine hides is beneficial when the soaking is carried out with low or no mechanical effect, since the cleaning of the hide is faster. This is the case of dried or dried-salted hides, in which the first soaking is usually performed in pits or paddles. Ultrasound must impact directly on the hides and on the float to achieve greater soaking efficiency and thus the cleaning of the hides becomes faster.

According to the results obtained in the tests on goatskins (section 3.2), it can be concluded that the effect of ultrasound follows the same general rules for goatskins than for bovine hides. The use of ultrasound increases the soaking speed versus the working systems with low or without mechanical effect (pits and paddles). In the cases where soaking is performed with a high mechanical effect (drumming), the use of ultrasound offers no advantages. However, the environmental benefit of using ultrasound could be very important in this case. Usually, in the soaking of goatskins, 20L of water per kilogram of dried skin are used [7]. This is the necessary amount for the blades of the paddles to proper agitate the soaking float. Ultrasound could replace the blades of the paddles and only half the water volume would be necessary. According to FAO [24], in 2011 the production of dried goatskins was approximately 300 thousand tons. Soaking with ultrasound would have meant savings of 3 million cubic meters of water. Logically, sheepskins will follow the same or very similar performance. Actually, the savings would be even higher because wool felting would be avoided. 40L of water per kilogram of dried sheepskin are commonly used in the soakings [7] to avoid as much as possible the contact in movement of the skins in the float, which may cause felting. In some cases, depending on its quality or price, the wool is destroyed during the tanning process and then, only 20L per kilogram of dried sheepskin [7] are used in the soakings. In both cases, the use of ultrasound would allow reducing the floats by half, because no movement of the skins would then be needed in the soaking operation. Although it is impossible to quantify the percentages in each case and thus an estimate of the potential water savings, we will assume that the wool in 50% of the tanned sheepskins and lambskins would be recovered and that in the other 50% would be destroyed. This is probably a conservative estimate, but it may give a rough idea of the magnitude of the potential water savings. According to FAO [24], in 2011 the production of dried sheepskins and lambskins was approximately 400 thousand tons. Soaking with ultrasound would have meant savings of 6 million cubic meters of water. Clearly, in this case the environmental benefit is very important. 9 (or probably more) million cubic meters of water per year could be saved. We must bear in mind that in most countries where the tanning industry is significant, water is increasingly becoming a necessary and insufficient good due to rises in population and industrial growth.

Table 5 describes, qualitatively, the main advantages and disadvantages of the use of ultrasound in the soaking. A quantitative analysis cannot be done without taking into account the skin characteristics (type, race, thickness, preservation, etc.), the work system applied and the final item being sought.

5. Conclusions

The results obtained in this work indicate that the use of ultrasound in the soaking is a valid alternative if the objective is to perform the process without using a drum. Very positive results for salt-dried bovine hides and salt-dried goatskins have been obtained. Results indicate that the application of ultrasound in the soaking, especially in goatskins is a good alternative to consider (versus systems currently employed) to increase cleaning and ensure proper skin soaking in less time or with lower water consumption. On the other hand, when the soaking can be carried out with a high mechanical effect, as it is in the case of wet-salted bovine hides, the use of the drum instead ultrasound is better. The results show that the use of ultrasound could be very suitable in the sheepskins soaking. Future research on this subject could provide significant environmental improvements.

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www.fao.org/economic/est/publications/hides-skins-publications/en/ (accessed 16/04/2012)

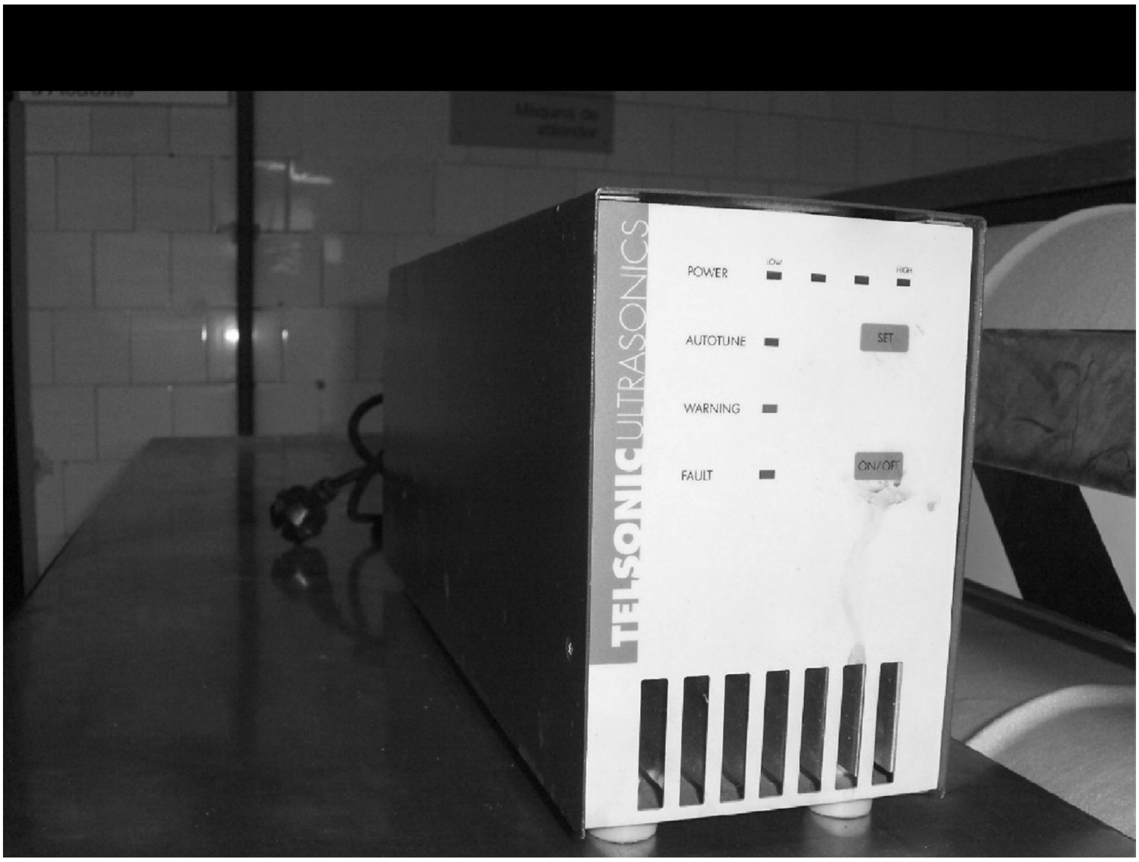

Fig. 1. Generator.

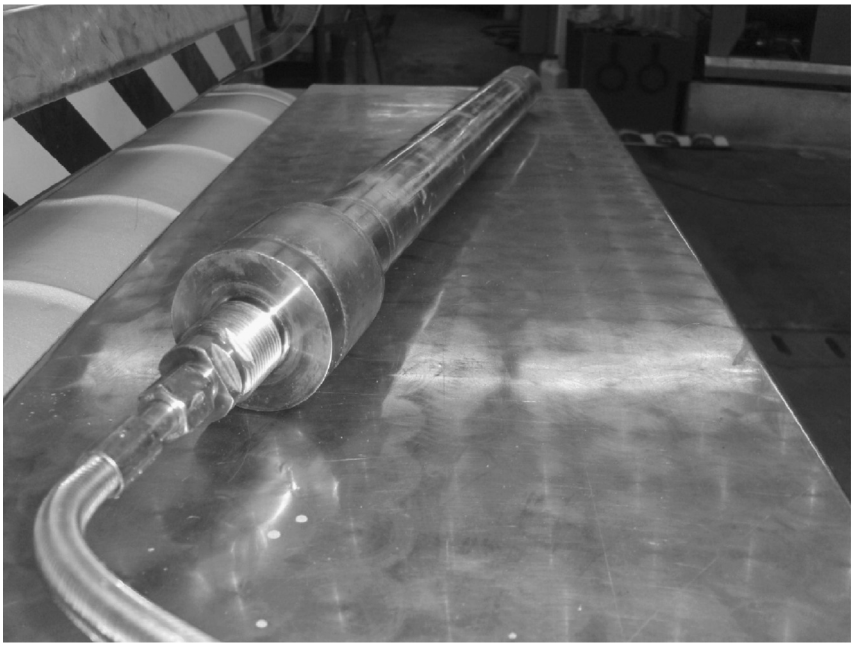

Fig. 2.
Transmitter.

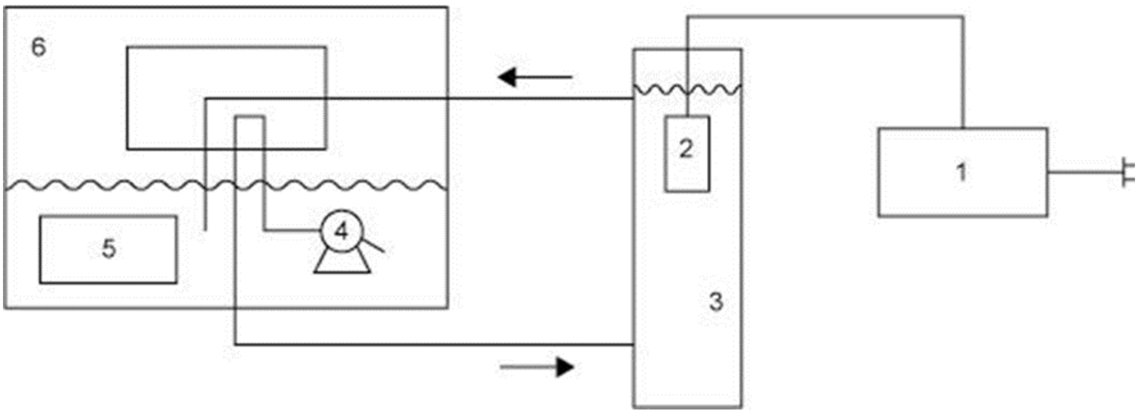

Fig. 3. Setup diagram. 1: US generator; 2: US transmitter; 3: Steel casing; 4: Pump; 5: Hide; 6: Drum.

Fig. 4. Photo of experimental setup.

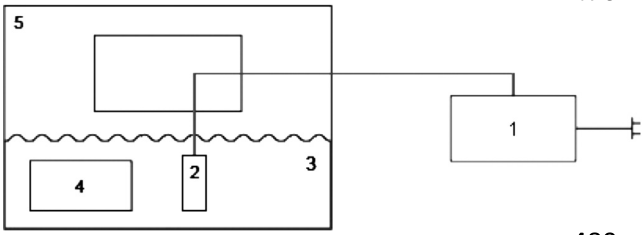

Fig. 5. Setup diagram. 1: US Generator; 2: US Transmitter; 3: Float; 4: Hide; 5: Drum.

Fig. 6. Photo of experimental setup.

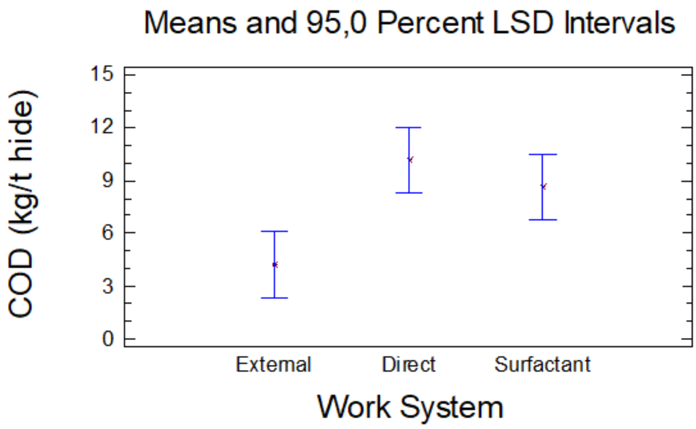

Fig. 7. Graphical representation of the analysis of variance.

Table 1

Time for each work system (goatskins)

Work system	Time
External	40 min. US + 30 min. S
Direct	60 min. US + 10 min. S
Surfactant	40 min. US + 15 min. S
Without US	70 min

Note: US = with ultrasound help; S = statically.

Table 2

Results on bovine hides.

Work system	Part of the hide	H_2O absorbed (%)	Conductivity mS/cm·t hide	COD kg/t hide	SS kg/t hide
External	Belly	23.09	1927	4.17	3.29
	Neck	24.77	1310	3.00	2.51
	Butt	28.63	2491	5.60	5.03
Direct	Belly	30.54	1805	8.57	4.09
	Neck	30.75	1642	7.74	5.04
	Butt	27.06	2136	14.19	7.77
Surfactant	Belly	28.67	1671	5.71	3.25
	Neck	28.45	1436	8.74	5.21
	Butt	32.04	2555	11.48	6.15
Static (Control)	Belly	24.33	1493	5.07	2.97
	Neck	28.67	1463	4.83	2.90
	Butt	29.00	2120	9.07	4.17

Table 3

Analysis of Variance for COD - Type III Sums of Squares.

Source	Sum of Squares	Df	Mean Square	F-Ratio	P-Value
MAIN EFFECTS					
A:Part	34,0289	2	17,0144	6,19	0,0596
B:Work System	56,2289	2	28,1144	10,23	0,0267
RESIDUAL	10,9911	4	2,74778		
TOTAL (CORRECTED)	101,249	8			

All F-ratios are based on the residual mean square error.

Table 4

Results on goatskins

Soaking without mechanical effect

Work System	H_2O absorbed (%)	Conductivity (mS/cm·t skin)	COD (kg/t skin)	SS (kg/t skin)
Static (Control)	144.44	9957	9.18	5.23

Soaking with ultrasound: Percentage difference compared with soaking without mechanical effect

Work System	H_2O absorbed (%)	Conductivity (%)	COD (%)	SS (%)
External	16.3	-9.6	67.9	29.6
Direct	1.7	9.3	101.2	48.2
Surfactant	11.0	-2.59	162.4	87.4

Table 5

Use of ultrasound in soaking: Main advantages and disadvantages.

Water consumption	The water savings can be important, especially in the goatskins and sheepskins soaking (≥50%)
Chemicals	Not chemicals are needed (Direct System)
Time	Time reductions are important in goatskins (36% minimum)
Energy consumption	Soaking using ultrasound requires more energy consumption than soaking statically. Power consumption is acceptable at industrial level (4.8 W/L float)